

**COMPARISONS OF ASTM METHODS
REFERENCED IN THE RCRA REGULATIONS AT 40 CFR 261.21(a)(1)
WITH NEWER EDITIONS OF THE METHODS**

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Introduction

The U.S. Environmental Protection Agency (the Agency or EPA) plans to publish a proposed rule (to be known as the Methods Innovation Rule or MIR) which will include the replacement of ASTM standards D 93-80 and D 3278-78 with D 93-99c and 3278-96, respectively, in the RCRA regulations at 40 CFR 261.21(a)(1). This document includes section-by-section comparisons of the newer methods with their counterpart older versions. Section differences are summarized.

Comparison of ASTM D 3278-78, Flash Point of Liquids by Setaflash Closed Tester and ASTM D 3278-96, Flash Point of Liquids by Small Scale Closed-Cup Apparatus

Purpose of Comparison

The Agency is considering the replacement of a reference to ASTM D 3278-78, Flash Point of Liquids by Setaflash Closed Tester, with ASTM D 3278-96, Flash Point of Liquids by Small Scale Closed-Cup Apparatus, in the RCRA regulations for the ignitability characteristic at 40 CFR 261.21(a)(1) and in SW-846 Method 1020. ASTM D 3278-96 is a more current edition of the procedure than ASTM D 3278-78. The purpose of this comparison of ASTM D 3278-78 and ASTM D 3278-96 is to document and summarize section-by-section differences between the methods.

Summary of Findings

Most of the new information found in the 1996 version represents minor clarifications of the procedure. Test Method A (Flash/No Flash) of the 1996 version is essentially the same as Test Method A of the 1978 version. New material was incorporated into Test Method B (Finite Flash Point) of the 1996 version to emphasize the need to perform duplicate determinations of the flash point. However, the method is essentially the same test found in the 1978 version.

Details regarding the comparison follow.

Results of Line-by-Line Comparison

1. Scope

Section 1.1 of the 1996 version is similar to Sections 1.1 and 1.2 of the 1978 version.

Section 1.2 of the 1996 version has material from Section 1.4 of the 1978 version and new material regarding the use of the test results for fire-hazard or fire-risk assessments.

Section 1.3 of the 1996 version contains new material that addresses safety concerns.

Note 1 of the 1996 version is identical to Note 1 of the 1978 version.

Notes 2 through 5 of the 1996 version contain new material.

2. Referenced Documents

This section is essentially identical to Section 2 of the 1978 version, with minor editorial changes, and also includes ISO standards.

3. Terminology

This section of the 1996 version provides a definition of flash point, which was not included in the 1978 version.

4. Summary of Test Method

Sections 4.1 and 4.2 in the 1996 version are similar to Sections 3.1 and 3.2 of the 1978 version. The 1996 version replaced the term "Setaflash Tester" with "small scale closed-cup apparatus." The two terms, however, are used to describe the same apparatus depicted in Annex A1.

5. Significance and Use

Section 5 of the 1996 version contains new information regarding the various uses of the flash point test. This section was not included in the 1978 version.

6. Apparatus

Sections 6.1 through 6.5 of the 1996 version are essentially identical to Sections 4.1 through 4.5 of the 1978 version.

7. Reagents and Materials

Section 7.1 and Sections 7.3 through 7.4 of the 1996 version are nearly identical to Sections 5.1 through 5.4 of the 1978 version, respectively.

Section 7.2 of the 1996 version adds n-butanol as a second optional reference standard. The 1978 Setaflash method includes one calibration standard, p-xylene. n-Butanol was added as a second standard to be consistent with the range of flash point used by "U.S. regulatory agencies in defining flammable liquids."

8. Sampling

Section 8.1 of the 1996 version is identical to Section 6.1 of the 1978 version.

Note 6 of the 1996 version is identical to Section 6.2 of the 1978 version.

Note 7 of the 1996 version contains new material on sample storage procedures to prevent the loss of volatile material from the sample.

9. Preparation of Apparatus

Sections 9.1 through 9.5 of the 1996 version are similar to Sections 7.1 through 7.5 of the 1978 version. The only differences are that Section 9.5 of the 1996 version mentions the use of n-butanol as a second option of a reference standard, and Section 9.2 of the 1996 version makes an editorial correction changing 9 °F to 10 °F.

TEST METHOD A - FLASH/NO FLASH

10. Procedure - Ambient to 110°C (230°F)

Sections 10.1 through 10.7 in the 1996 version are essentially identical to Sections 8.1 through 8.7 of the 1978 version.

Notes 8 and 9 of the 1996 version are essentially identical to the Section 8 Notes 2 and 3 of the 1978 version.

Note 10 of the 1996 version is somewhat different from the corresponding note (note 4) in the 1978 version. The note in the 1996 version recommends that the nozzle of the ignition device intersect the plane of the underside of the cover when inserted. The note in the 1978 version states that the nozzle of the ignition device shall be 1 ± 1 mm above the underside of the cover when inserted into the test cup.

Note 11 of the 1996 version is identical to Note 5 of the 1978 version.

11. Procedure - 0°C (32°F) to Ambient

Section 11.1 through 11.4 of the 1996 version are essentially the same as Sections 9.1 through 9.4 of the 1978 version, respectively.

Notes 12 and 13 of the 1996 version are essentially identical to Section 9.2.1 and Note 6 of the 1978 version, respectively.

TEST METHOD B - FINITE FLASH POINT

12. Procedure - Ambient to 110°C (230°F)

Sections 12.1.1 and 12.1.2 of the 1996 version are essentially identical to Sections 10.1 and 10.2 of the 1978 version.

Sections 12.2 and 12.3 of the 1996 version are essentially the same as Sections 10.3 and 10.3.1 of the 1978 version, respectively. Section 12.3 has a typographical error, it refers the reader to Note 4 instead of Note 14.

Note 14 of the 1996 version is identical to Note 7 of the 1978 version.

Section 12.3.1 of the 1996 version is similar to Section 10.3.2 of the 1978 version; the wording differs slightly.

Section 12.3.1.1 of the 1996 version contains new material requiring the operator to repeat the determination of the flash point (12.3.1) with a new specimen, and calculate the corrected mean temperature. The 1978 version of the method states the need for duplicate determinations of the flash point in its Section 14. In that section the operator is required to report the mean of duplicate determinations to the nearest 0.5 °C, provided that the values do not differ by >1 °C.

Section 12.4 of the 1996 version is essentially the same as Section 10.4.1 of the 1978 version.

Section 12.4.1 of the 1996 version contains new material requiring the operator to repeat the determination of the flash point (12.3.1) with a new specimen, and calculate the corrected mean temperature. The 1978 version of the method addresses the need for duplicate determinations of the flash point in its Section 14. In that section the operator is required to report the mean of duplicate determinations to the nearest 0.5 °C, provided that the values do not differ by >1 °C.

13. Procedure - 0 °C (32 °F) to Ambient

Sections 13.1.1 through 13.3.1 of the 1996 version are essentially the same as Sections 11.1 through 11.4.2 of the 1978 version, respectively.

Section 13.3.1.1 of the 1996 version contains new material requiring the operator to repeat the determination of the flash point (13.3.1) with a new specimen, and calculate the corrected mean temperature. The 1978 version of the method addresses the need for duplicate determinations of the flash point in its Section 14. In that section the operator is required to report the mean of duplicate determinations to the nearest 0.5 °C, provided that the values do not differ by >1 °C.

Section 13.4 of the 1996 version is essentially identical to Section 11.5.1 of the 1978 version.

Section 13.4.1 of the 1996 version contains new material requiring the operator to repeat the determination of the flash point (13.3.1) with a new specimen, and calculate the corrected mean temperature. The 1978 version of the method states the need for duplicate determinations of the flash point in its Section 14. In that section the operator is required to report the mean of duplicate determinations to the nearest 0.5°C, provided that the values do not differ by >1 °C.

14. Clean-up of Apparatus and Preparation for New Test

Sections 14.1 through 14.4 of the 1996 version are identical to Sections 12.1 through 12.4 of the 1978 version, respectively.

Notes 15 and 16 of the 1996 version are identical to Notes 8 and 9 of the 1978 version.

15. Correction for Barometric Pressure

Sections 15.1 and 15.2 of the 1996 version are essentially the same as Sections 13.2 and 13.1 of the 1978 version, respectively.

16. Report

Sections 16.1 and 16.2 of the 1996 version are essentially identical to Sections 14.1 and 14.2 of the 1978 version, respectively.

17. Precision and Bias

Section 17.1 of the 1996 version contains new material regarding the precision and bias of Test Method A. The 1996 version states that there are no precision data for the flash/no flash test since the result only indicates whether or not the sample flashes at the required flash point. No information about the precision or bias of Test Method A was given in the 1978 version.

Sections 17.2 through 17.4 of the 1996 version are essentially identical to Sections 15.1 through 15.3 of the 1978 version, respectively.

Sections 17.5 and 17.6 of the 1996 version are new, specifying two types of materials for which precision data are not available.

Section 17.7 of the 1996 version is new, indicating that it is inappropriate to estimate the bias of this test.

18. Keywords

This section contains a list of keywords in the 1996 version. Keywords are not given in the 1978 version.

19. Annexes

Annex A1 and A2 of the 1996 version are essentially identical to the corresponding annexes in the 1978 version.

Annex A3 of the 1996 version has similar information to that in Annex A3 of the 1978 version as well as new material regarding the use of n-Butanol as a reference standard.

Annex A4 of the 1996 version is essentially identical to the corresponding annex in the 1978 version.

Annex A5 of the 1996 version contains all new material regarding the testing of organic peroxides.

Comparison of ASTM D 93-80 and ASTM D 93-99c Flash Point by Pensky-Martens Closed Cup Tester

Purpose of Comparison

The Agency is considering the replacement of a reference to ASTM D93-80 with ASTM D93-99c, Flash Point by Pensky-Martens Closed Cup Tester, in RCRA regulations for the ignitability characteristic at 40 CFR 261.21(a)(1) and in SW-846 Method 1010. ASTM D93-99c is a most more recent version than D93-80. The purpose of a comparison of ASTM D 93-80 and ASTM D 93-99c before the replacement is to identify any substantive differences. Substantive differences might affect overall how many wastes are identified as hazardous based on the ignitability characteristic and might make the procedure significantly much more costly to perform.

Summary of Findings

The line-by-line comparison of the 1980 (ASTM D 93-80) and 1999 (ASTM D 93-99c) versions of the Flash Point by Pensky-Martens Closed Cup Tester Methods revealed some differences. Most of the new information found in the 1999 version deals with the use of an automated apparatus for performing the test, some new instructions regarding sample collection, and the verification of apparatus performance by using certified reference materials and secondary working standards. The rest of the new material in the 1999 version offers precautionary information and additional guidance to aid the operator in performing the test.

New material of ASTM D93-99c in Procedure A, Determination of Flash Point of Fuel Oils, Lubricating Oils and Other Homogeneous Liquids, requires that the operator repeat the test with a new specimen at a lower temperature when a flash point is detected on the first application. A test repeat is not expensive or time consuming.

Similarly, Procedure A requires that an initial result be considered approximate and the test repeated when a flash point is detected at a temperature greater than 28 °C or less than 18 °C above the temperature of the first application of the ignition source. Its impact of this new requirement for the purpose of an ignitability determination under RCRA regulations is moot since 60°C is the standard temperature for such a determination.

The 1999 version also adds the use of certified reference materials (CRMs) and secondary working standards (SWSs) to check apparatus performance. The use of CRMs and SWSs provides some protection against false negative and false positive errors in the test.

Assuming that the 1980 version of the procedure is performed correctly in the first place, the addition of QC steps by the 1999 version is not significant.

Details regarding the comparison follow.

Results of Line-by-Line Comparison

1. Scope

In general, Section 1 of the 1999 version contains similar information to Section 1 of the 1980 version that is arranged in a different format.

Section 1.1 of the 1999 version contains new material, allowing the use of either a manual or an automated Pensky-Martens closed cup apparatus. In addition, Section 1.1 of the 1999 version specifies a temperature range of 40 to 360 °C for determination of the flash point of petroleum products. Section 6.1 of the 1980 version contained language allowing the use of an automated testing apparatus.

Note 1 of the 1999 version is new and states that the precision of the procedures above 250 °C (and 100 °C for residual fuels) has not been determined.

Section 1.2 of the 1999 version contains new information regarding the applicability of Procedure A for flash point determination of different types of substances.

Section 1.3 of the 1999 version is similar to Section 1.1 of the 1980 version; it also contains additional information regarding the applicability of Procedure B for flash point determination of different types of substances.

Note 2 of the 1999 version is almost identical to Section 1.2 of the 1980 version.

Section 1.4 of the 1999 version is similar to Note 2 of the 1980 version regarding the use of the test for detection of material contamination.

Section 1.5 of the 1999 version is similar to Section 1.4 of the 1980 version, with the exception that in the 1999 version of the method SI units (°C) are regarded as the standard and values are provided in °F for information purposes only.

Section 1.6 of the 1999 version contains new information advising the user to establish appropriate safety and health practices during use.

2. Reference Documents

The 1999 version contains a longer list of reference documents than the 1980 version, including for ISO standards.

3. Terminology

Sections 3.1.1, 3.1.1.1, and 3.1.1.2 of the 1999 version are essentially identical to Sections 5.1, 5.1.1, and 5.1.2 of the 1980 version, respectively.

Sections 3.1.2 and 3.1.3 of the 1999 version contain additional definitions.

4. Summary of Test Method

Section 4.1 of the 1999 version is similar to Section 3.1 of the 1980 version of the method, with the exception that the 1999 version specifies that the cup is to be filled with the sample to the level indicated by the filling mark and fitted with a cover of specified dimensions. In addition, Section 4.1 of the 1999 version specifies that the test specimen be heated and stirred by either of two procedures, A or B.

5. Significance and Use

Sections 5.1 and 5.2 of the 1999 version are nearly identical to Sections 4.1 and 4.2 of the 1980 version, respectively.

Note 3 of the 1999 version is nearly identical to Note 3 of the 1980 version.

Sections 5.3 of the 1999 version contains similar information to that found in Section 1.3 of the 1980 version.

Section 5.4 of the 1999 version contains new information claiming this method to be the only closed cup flash point test for temperatures up to 370 °C.

6. Apparatus

The 1999 version of the method discusses two types of apparatus: Manual Pensky-Martens Closed Cup Apparatus (Section 6.1) and Automated Pensky-Martens Closed Cup Apparatus (Section 6.2). Section 6.1 of the 1980 version contained language allowing the use of an automated testing apparatus.

Sections 6.1 and 6.2 of the 1999 version provide more detailed information about the apparatus than Section 6.1 of the 1980 version, but they all refer the reader to Annex A1 for description of apparatus, which is essentially identical to Annex A1 of the 1980 version.

Section 6.3 of the 1999 version contains similar information to Section 6.2 of 1980 version, but presented in tabular format.

Section 6.4 of the 1999 version presents new material describing the ignition source. This section refers the reader to Figure A1.4, which is the same figure found in the 1980 version.

Note 4 of the 1999 version provides new information regarding gas pressure.

7. Reagents and Materials

This is a new section in the 1999 version and it contains information on cleaning solvents.

8. Sampling

Section 8.1 of the 1999 version presents new material regarding how to obtain a sample.

Section 8.2 of the 1999 version has new material regarding how much sample is required for each test, and regarding the fullness of the sample container.

Section 8.3 of the 1999 version contains new information describing how to avoid the loss of volatile material from the sample.

Sections 8.4 of the 1999 version is similar to Section 8.3 of the 1980 version with the exception that Section 8.4 of the 1999 version notes that samples in leaky containers are suspect.

Section 8.5 of the 1999 version is similar to Section 8.1 of the 1980 version, but includes additional information regarding the handling of very viscous materials.

Section 8.6 of the 1999 version is nearly identical to Section 8.2 of the 1980 version of the method, except that the temperature given is 1 degree C higher.

Note 6 of the 1999 version contains precautionary information about the loss of volatile vapors.

Note 7 of the 1999 version contains new precautionary information about the loss of volatile vapors during heating of viscous samples.

Note 8 of the 1999 version is nearly identical to Note 5 of the 1980 version.

9. Preparation of Apparatus

Sections 9.1 and 9.2, and Note 9 of the 1999 version are similar to Section 7.1 of the 1980 version.

Note 10 of the 1999 version contains new material on performing the test under a hood for those samples whose vapors or products of pyrolysis are objectionable.

Section 9.3 of the 1999 version contains new material regarding the preparation of the apparatus for operation.

Note 11 of the 1999 version provides new information regarding gas pressure.

Section 9.4 of the 1999 version contains new material regarding the cleaning of the test cup and its accessories before the test.

Note 12 of the 1999 version contains new material on the use of cleaning solvents.

Note 13 of the 1999 version contains new precautionary information on the flammability of cleaning solvents.

10. Verification of Apparatus (Calibration and Standardization)

Section 10 of the 1999 version contains all new information regarding the verification of the apparatus' performance. The 1980 version of the method had no quality control procedures.

11. Procedure A

Sections 11.1 through 11.1.4 of the 1999 version are similar to Section 9.1 of the 1980 version. The 1999 version specifies that the temperature of the test cup and test specimen need to be at least 18 °C or 32 °F below the expected flash point before starting the test.

Notes 14 through 17 of the 1999 version contain new precautionary information for the operator performing the test.

Sections 11.1.5.1 and 11.1.5.2 of the 1999 version are similar to Sections 9.2 and 9.3 of the 1980 version, respectively, with some minor differences in the temperature range specified. In the 1980 version of the method, the flame is applied when the temperature is between 17 °C and 28 °C below expected flash point, while the new version specifies a temperature range of 18 °C to 28 °C.

Section 11.1.6 of the 1999 version is nearly identical to Note 6 of the 1980 version.

Section 11.1.7 of the 1999 version has new information on performing the test on those samples where the expected flash point temperature is not known. This new material provides more guidance to the operator.

Notes 18 and 19 of the 1999 version contain additional guidance for the operator.

Sections 11.1.8 and 11.1.9 of the 1999 version are essentially the same as Section 9.4 of the 1980 version.

Note 20 of the 1999 version a description regarding the point at which the sample is deemed to have flashed.

Section 11.1.10 of the 1999 version contains new information advising the user to repeat the test with a new specimen when a flash point is detected on the first application.

Section 11.1.11 of the 1999 version contains new information specifying that an initial result be considered approximate and the test repeated when a flash point is detected at a temperature greater than 28 °C or less than 18 °C above the temperature of the first application of the ignition source.

Section 11.1.12 of the 1999 version contains more guidance for the operator performing the test.

Note 21 of the 1999 version contains additional guidance for the operator.

Section 11.2 of the 1999 version contains information on performing the test using the automated apparatus. This information is similar to the information presented for the manual apparatus in Sections 11.1.1 through 11.1.8 in the 1999 version.

12. Procedure B

Sections 12.1.1 and 12.1.2 of the 1999 version contain more detailed information on the use of the manual apparatus in Procedure B than Section 10 of the 1980 version. This new material is similar to that in Sections 11.1.1 and 11.1.2 of the 1999 version for Procedure A.

Notes 22 through 25 of the 1999 version contain precautionary information for the operator similar to Notes 14 through 17 of the Procedure A 1999 version.

Sections 12.1.3 through 12.1.5 of the 1999 version are essentially identical to Section 10.1 of the 1980 version.

Sections 12.2 of the 1999 version contains new information on performing Procedure B using the automated apparatus. This information is similar to the information presented for performing Procedure A using the automated apparatus in Sections 11.2 of the 1999 version.

13. Calculation

Sections 13.1 and 13.2 of the 1999 version are similar to Sections 11.1 and 11.2 of the 1980 version, respectively.

Note 26 of the 1999 version is essentially the same as Note 7 of the 1980 version.

14. Report

Section 14.1 of 1999 version is essentially identical to Section 11.3 of 1980 version

15. Precision and Bias

Sections 15.1.1 and 15.1.2 of the 1999 version contain new values for repeatability and reproducibility for Procedure A.

Section 15.1.3 of the 1999 version contains new information regarding bias.

Section 15.1.4 of the 1999 version contains new information about relative bias for Procedure A. The 1999 version specifically states that statistical data did not reveal significant differences between the reproducibility variances of manual and automated Pensky-Martens flash point results.

Note 27 of the 1999 version contains additional information regarding precision data.

Section 15.1.5 of the 1999 version contains new information regarding a 1994 cooperative test program to obtain test precision data for Procedure A.

Sections 15.2.1 and 15.2.2 of the 1999 version are similar to Sections 12.2.1 and 12.2.2 of the 1980 version regarding the repeatability and reproducibility of Procedure B.

Sections 15.2.3 of the 1999 version contain new information regarding bias for Procedure B. The 1999 version specifically states that no bias has been determined for Procedure B.

Sections 15.2.4 and 15.2.5 of the 1999 version contain new information regarding the development of test precision data for Procedure B.

Note 28 of the 1999 version contains additional information regarding precision data.

16. Keywords

This section of the 1999 version contains a list of keywords. Keywords are not given in the 1980 version.

17. Annexes

Annexes A.1, and A.2 of the 1999 version are essentially identical to the corresponding annexes in the 1980 version. Annex A.1 of the 1999 version contains more detailed information about the stirring device than the corresponding annex in the 1980 version. However, both annexes refer the reader to figure A1.4, which is identical in both versions of the method. Annex A.1 of the 1999 version also contains new information regarding the use of electric ignitors.

Annex A3 provides slightly different thermometer specifications from those in the 1980 version, and a new "Table A3.4 Specifications for Medium-Range Pensky Martins".

Annex 4 of the 1999 version contains new information regarding certified reference materials and secondary working standards.

Annexes are newly identified as "(Mandatory Information)"

Comparison of ASTM D 93-99c and ASTM D 93-00 Flash Point by Pensky-Martens Closed Cup Tester

The following is a summary of the main differences between ASTM D 93-99c and D 93-00.

1. In section 2. Referenced Documents, the following document was added to the '00 version: E 502 Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods.
2. Paragraph 8.2 was revised to describe the different volumes required in the containers for each sample type. The sentence in 99c "The sample container shall be at least 85% full" was replaced with "When obtaining a sample of residual fuel oil, the sample container shall be from 85 to 95% full. For other types of samples, the size of the container shall be chosen such that the container is not more than 85% full or less than 50% full prior to any sample aliquot being taken."
3. Text was added to the '00 version as paragraph 8.3, allowing successive specimens from the sample container and minimum sample container volumes. The new text reads: "Successive test specimens can be taken from the same sample container. Repeat tests have been shown to be within the precisions of the method when the second specimen is taken with the sample container at least 50% filled. The results of flash point determinations can be affected if the sample volume is less than 50% of sample container capacity."
4. Paragraph 11.1.1 was revised to ensure that the sample container volumes are as specified in 8.2. The following sentence now precedes the original paragraph: "Ensure that the sample container is filled to the volume capacity requirement specified in 8.2."
5. A warning note was added to version '00 after paragraph 11.1.8 about halogenated hydrocarbons and the flash point interference.
6. Paragraph 12.1.1 was revised to ensure that the sample container volumes are as specified in 8.2. The following sentence now precedes the original paragraph: "Ensure that the sample container is filled to the volume capacity requirement specified in 8.2."
7. Table A3.2, column 14 (which refers to Bulb/OD E) , Row 2 (which refers to ASTM 9C-62) states "[gnE] stem" while the '00 version states "not greater than stem".
8. Appendix X1 Flash Point Masking Phenomenon was added.
9. Appendix X2 Flash Point Test and Flammability of Mixtures was added.